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FYI-13-01703	84130000006	2/12/13

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Shin-Etsu MicroSi, Inc.

February 6, 2013

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TSCA Confidential Business Information Center (7407M)
EPA East - Room 6428 Attn: FYI
U.S. Environmental Protection Agency
1200 Pennsylvania Avenue, NW
Washington, DC 20460-0001

FYI-13-01703

RE: TSCA FYI submission of Biodegradation study of RESIN-A2M

To Whom It May Concern,

Shin-Etsu MicroSi is providing the attached Biodegradation study of RESIN-A2M as an "FYI" submittal to the EPA.

Study Objective: To evaluate the biodegradability of RESIN-A2M by microorganisms.

Chemical identity:

Chemical name: Formaldehyde, polymer with dimethylphenol and methylphenol, acetate 6-diazo-5,6-dihydro-5-oxo-1-naphthalenesulfonate.

Shin-Etsu MicroSi alias name: RESIN-A2M

CAS RN: 1361249-72-0

Please contact me at 480-893-8898 or jedmonds@microsi.com for questions regarding this submittal.

Thank you,

8413000000006

Jim Edmonds, President

February 6, 2013
Date

CONTAINS NO CBI

www.MICROSI.com
480.893.8898 | FAX 480.893.8637
10028 S. 51ST STREET | PHOENIX, AZ 85044

CERI

Receipt number	632-12-S-5857
Study number	15857

FINAL REPORT

Biodegradation study of RESIN-A2M

December, 2012

Chemicals Evaluation and Research Institute, Japan, Kurume

STATEMENT

Chemicals Evaluation and
Research Institute, Japan, Kurume

Sponsor Shin-Etsu Chemical Co., Ltd.

Title Biodegradation study of RESIN-A2M

Study number 15857

I, the undersigned, hereby declare that this report provides a correct English translation of the Final Report (Study No. 15857, issued on December 28, 2012).

Date January 28, 2013

Translator Kotaro Okuzono

Kotaro Okuzono

GLP STATEMENT

Chemicals Evaluation and
Research Institute, Japan, Kurume

Sponsor Shin-Etsu Chemical Co., Ltd.

Title Biodegradation study of RESIN-A2M

Study number 15857

The study described in this report was conducted in compliance with the following GLP principles:
"Standard Concerning Testing Facility Relating to New Chemical Substances" (March 31, 2011; No. 0331-8, Pharmaceutical and Food Safety Bureau, Ministry of Health, Labour and Welfare; March 29, 2011, No. 6, Manufacturing Industries Bureau, Ministry of Economy, Trade and Industry; No. 110331010, Environmental Policy Bureau, Ministry of the Environment, Japan)

This final report reflects the raw data accurately and it has been confirmed that the test data are valid.

Date December 28, 2012

Study Director Signed in original
Kotaro Okuzono

QUALITY ASSURANCE STATEMENT

Chemicals Evaluation and
Research Institute, Japan, Kurume

Sponsor Shin-Etsu Chemical Co., Ltd.

Title Biodegradation study of RESIN-A2M

Study number 15857

I assure that the final report accurately describes the test methods and procedures, and that the reported results accurately reflect the raw data of the study.

The inspections and audits of this study were carried out and the results were reported to the Study Director and the Test Facility Management by Quality Assurance Unit as follows.

Item of inspection / audit	Date of inspection / audit	Date of report to Study Director and Test Facility Management
Study plan draft	August 3, 2012	August 6, 2012
Study plan	August 6, 2012	August 6, 2012
At the start of cultivation	August 7, 2012	August 7, 2012
Recovery test	August 21, 2012	August 21, 2012
At the end of cultivation	September 4, 2012	September 4, 2012
Amendment to study plan 1	October 3, 2012	October 3, 2012
Amendment to study plan 2	December 27, 2012	December 27, 2012
Raw data and final report draft	December 28, 2012	December 28, 2012
Final report	December 28, 2012	December 28, 2012

Date December 28, 2012

Head of Quality Assurance Unit Signed in original
Ryuichiro Mizuguchi

CONTENTS

1. Title

Biodegradation study of RESIN-A2M

2. Sponsor

Name	Shin-Etsu Chemical Co., Ltd.
Address	6-1, Ohtemachi 2-Chome, Chiyoda-ku, Tokyo 100-0004, Japan

3. Test facility

Name	Chemicals Evaluation and Research Institute, Japan, Kurume (CERI Kurume)
Address	3-2-7 Miyanojin, Kurume-shi, Fukuoka 839-0801, Japan

4. Objective

This study was aimed at evaluating the biodegradability of RESIN-A2M by microorganisms.

5. Test method

"Method for Testing the Biodegradability of Chemical Substances by Microorganisms" stipulated in the "Testing Methods for New Chemical Substances" (March 31, 2011, No. 0331-7, Pharmaceutical and Food Safety Bureau, Ministry of Health, Labour and Welfare; March 29, 2011, No. 5, Manufacturing Industries Bureau, Ministry of Economy, Trade and Industry; No. 110331009, Environmental Policy Bureau, Ministry of the Environment, Japan; April 2, 2012 partial revision, No. 0402-1, Pharmaceutical and Food Safety Bureau, Ministry of Health, Labour and Welfare; March 28, 2012, No. 2, Manufacturing Industries Bureau, Ministry of Economy, Trade and Industry; No. 120402001, Environmental Policy Bureau, Ministry of the Environment, Japan)

However, some of this study deviated from the above second method. Contents of the deviation were described in "14. Validity of the test conditions".

6. GLP principles

"Standard Concerning Testing Facility Relating to New Chemical Substances" (March 31, 2011; No. 0331-8, Pharmaceutical and Food Safety Bureau, Ministry of Health, Labour and Welfare; March 29, 2011, No. 6, Manufacturing Industries Bureau, Ministry of Economy, Trade and Industry; No. 110331010, Environmental Policy Bureau, Ministry of the Environment, Japan)

7. Dates

Study initiation date	August 6, 2012
Experimental starting date	August 7, 2012
Experimental completion date	September 4, 2012
Study completion date	December 28, 2012

8. Storage of test item, raw data, etc.

The study plan (original), the final report (original), the raw data, documents concerning the study presented by the sponsor, other reports, and the test item are stored in the archives of this laboratory.

The storage period is 10 years after the study completion date. In case of receipt of the notice specified under Clause 1 or Clause 2 of Article 4; Clause 2, Clause 3, or Clause 8 of Article 5; Clause 3 of Article 10; or Clause 2 of Article 14 of the "Law Concerning Examination and Regulation of Manufacture etc. of Chemical Substances," these items are stored for 10 years from that date. After 10 years from the study completion date, the storage period is discussed with the sponsor. The stability of the test item is not confirmed during the storage period.

The management of the test item and raw data, etc. after the storage period (continue, reject, or return) is discussed with the sponsor.

9. Personnel

Study Director	Kotaro Okuzono (Section 2)
Study personnel (Operation of biodegradation test)	Ayaka Maeda
Staff for cultivation of activated sludge	Noriko Miyaura

10. Approval of final report

Date December 28, 2012

Study Director Signed in original
Kotaro Okuzono

11. Summary

Conditions of cultivation

Concentration of test item	83.3 mg/L
Concentration of activated sludge	30 mg/L (as the concentration of suspended solid)
Volume of test solution	300 mL
Cultivation temperature	25±1°C
Cultivation duration	28 days (under dark conditions)

Measurement and analysis for calculation of percentage biodegradation

a) Measurement of biochemical oxygen demand (BOD) with a closed system oxygen consumption measuring apparatus

b) Determination of test item by high-performance liquid chromatography (HPLC)

Results

		Sludge + test item			
		Vessel No. 1	Vessel No. 2	Vessel No. 3	Average
Percentage biodegradation by BOD	%	16	12	15	14
Percentage biodegradation of test item (HPLC)	%	1	0	3	2

Conclusion

The test item was not biodegraded under the test conditions of this study.

12. Test materials

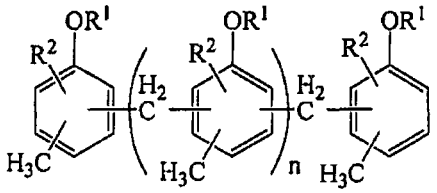
12.1 Test item

a) Chemical name etc.

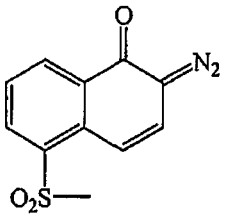
Chemical name	Formaldehyde, polymer with dimethylphenol and methylphenol, acetate 6-diazo-5,6-dihydro-5-oxo-1-naphthalenesulfonate
Another name	RESIN-A2M
CAS number	1361249-72-0

b) Chemical structure etc.

Structural formula



R^1= H or $CH_3C(=O)$— or



R^2= H or CH_3

Molecular weight	Number average molecular weight (Mn)	1386
	Weight average molecular weight (Mw)	8745
	Percentage of molecular weight < 800(%)	11.575
	Percentage of molecular weight < 1000(%)	13.894

c) Test sample

Purity of test item	82.7%
Impurity	Methyl isobutyl ketone 17.3%(GC)
Supplier	Shin-Etsu Chemical Co., Ltd.
Lot number	HN174

The test item concentration was corrected by its purity.

d) Physicochemical properties

Water solubility	Insoluble (visual)
Appearance	Brown powder
Stability	Photodegradable

Solubility to solvent

Solvent	Solubility	Stability in solvent
Acetone	≥500 g/L (visual)	Stable

e) Storage conditions

The test sample was stored in a cold and dark storage place.

f) Identification and stability of test item

The infrared (IR) spectrum of the test item measured at this laboratory was confirmed to be identical to that provided by the sponsor (see Fig. 5 and Reference 1).

The stability of the test item was confirmed by comparing the IR spectrum of the test item after the completion of the experiment with that before the start of the experiment (see Fig. 5).

g) Safety

In order to avoid inhalation and contact with the skin and eyes, chemically resistant gloves, mask, glasses, and white coats were worn when handling all chemicals. The test sample was handled under the yellow fluorescent light (light wavelength of below 500 nm was screened-out).

12.2 Reference item

a) Chemical name etc.

Chemical name	Aniline
CAS number	62-53-3

b) Supplier and lot number

Supplier	Wako Pure Chemical Industries
Lot number	DCG6435

12.3 Activated sludge

On-site sludge sampling was carried out at 10 locations in Japan (samples were from surface water and surface soil of rivers, lake, and inland seas; return sludges from sewage plants). Activated sludge, which was prepared and controlled in this laboratory according to the test method described in Section 5, was used in this study (sampling period: May, 2012, initiation date of use: June 18, 2012). The activated sludge, which was cultivated for 19 hours after the synthetic sewage was added, was used. The synthetic sewage was prepared according to the following method; glucose, peptone, and potassium dihydrogenphosphate were dissolved in purified water, and the pH of the solution was adjusted to 7.0±1.0.

13. Performance of the biodegradability test

13.1 Preparations for test

a) Decision of additive amount of activated sludge

Additive amount of activated sludge into the test vessel was 2.77 mL on the basis of the concentration of suspended solid in the activated sludge which was determined by the following methods;

Method	In accordance with Japanese Industrial Standards (JIS) K 0102-2008 Section 14.1
Date	August 6, 2012
Result	3250 mg/L

b) Preparation of basal culture medium

The basal culture medium (2 L) was prepared by the same method as follows; purified water (The Japanese Pharmacopoeia, Takasugi Pharmaceutical Co., Ltd.) was added to each 3 mL aliquot of solutions A, B, C and D, which are described in JIS K 0102-2008 Section 21, in order to prepare 1 L of solution. The pH of this solution was then adjusted to 7.0.

c) Validity of activated sludge

Aniline was used as a reference item in order to confirm that the sludge was sufficiently active.

13.2 Preparation of test solutions

The following test solutions were prepared and cultured under the conditions described in Section 13.3.

a) Addition of test item or aniline

1) Test solution (water + test item) (n=1, Vessel No. 5)

In one test vessel, 30 mg of the test sample (25 mg of test item) was accurately weighed by an electronic analytical balance and added to 300 mL of purified water (the concentration of the test item was 83.3 mg/L).

2) Test solution (sludge + test item) (n=3, Vessel Nos. 1, 2 and 3)

In each test vessel, 30 mg of the test sample (25 mg of test item) was accurately weighed by an electronic analytical balance and added to the basal culture medium [the volume subtracting the volume (2.77 mL) of activated sludge from 300 mL] (the concentration of the test item was 83.3 mg/L).

3) Test solution (sludge + aniline) (n=1, Vessel No. 6)

In one test vessel, 29.5 μL (30 mg) of aniline was taken out by microsyringe and added to the basal culture medium [the volume subtracting the volume (2.77 mL) of activated sludge from 300 mL], so that the concentration of aniline reached 100 mg/L.

4) Test solution (control blank) (n=1, Vessel No. 4)

In one test vessel, nothing was added to the basal culture medium [the volume subtracting the volume (2.77 mL) of activated sludge from 300 mL].

b) Inoculation of activated sludge

The activated sludge was added to each test vessel described in 2), 3) and 4), so that the concentration of the suspended solid reached 30 mg/L.

13.3 Instruments and conditions of cultivation

a) Instruments for cultivation

Closed system oxygen consumption measuring apparatus

Temperature controlled bath (containing a measuring unit) AI-0001 (Asahi Techneion)

Data sampler OM7000A (Ohkura Electric)

Vessel: Glass vessel (covered with aluminum foil)

Absorbent for carbon dioxide: Soda lime No.1 (for absorption of carbon dioxide, Wako Pure Chemical Industries)

b) Conditions of cultivation

Cultivation temperature	25±1°C
Cultivation duration	28 days (under dark conditions)
Stirring method	Each test solution was stirred by a stirrer.

c) Room

Apparatus room 1A

13.4 Observation and measurement of test conditions

a) Observation of test solution

During the cultivation period, the appearance of the test solution was observed once a day.

b) Measurement of biochemical oxygen demand (BOD)

During the cultivation period, BOD of the test solutions was measured continuously by a closed system oxygen consumption measuring apparatus. The cultivation temperature was measured and recorded once a day.

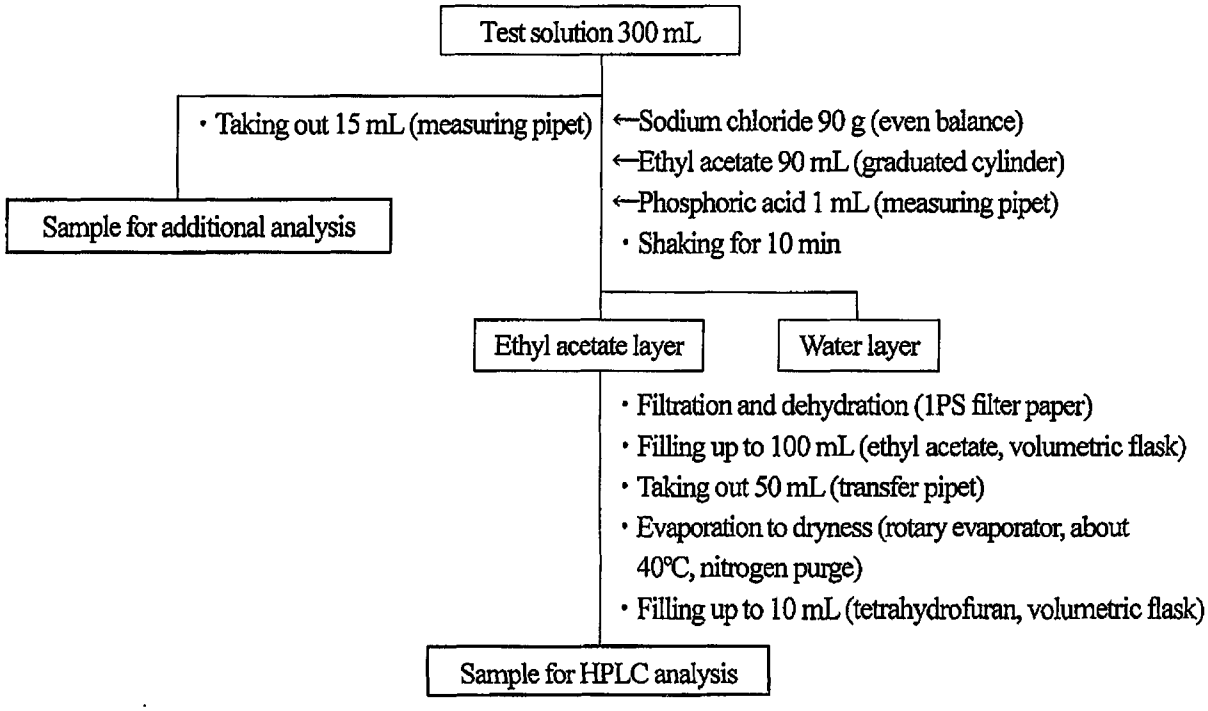
13.5 Analysis of test solution

After the end of the cultivation, the test item in the test solutions was determined.

Dissolved organic carbon (DOC) was not determined because the test item was not dissolved in the test solution in the preliminary test. The pH of the test solution (water + test item) and the test solutions (sludge + test item) was measured under the yellow fluorescent light.

13.5.1 Pretreatment of test solutions for analysis

The test solution (water + test item), the test solutions (sludge + test item), and the test solution (control blank) were pretreated to prepare samples for high-performance liquid chromatography (HPLC) analysis of the test item and additional analysis as follows: The sample for additional analysis was not analyzed because the test item was not converted. The pretreatment was performed under the yellow fluorescent light.



13.5.2 Determination of test item

The test item was analyzed quantitatively with HPLC.

a) Determination method

The test item was determined with absolute calibration curve method using one concentration of standard solution.

In order to confirm the validity of this determination method, the calibration curve was made using three concentrations of standard solution, 310, 620, and 1240 mg/L (see Fig. 2). As a result, the regression line of the calibration curve was a straight line from the origin, therefore the validity was confirmed.

b) Analytical conditions

Instrument	High-performance liquid chromatograph	
Pump	LC-10ADvp	(Shimadzu)
Differential refractive index detector		
	RI-104	(SHOWA DENKO)
Column oven	CTO-10ACvp	(Shimadzu)
Auto injector	SIL-20A	(Shimadzu)
System controller	SCL-10Avp	(Shimadzu)
Degasser	DGU-12AM	(Shimadzu)
Column	Shodex GPC KF-403HQ (250 mm × 4.6 mm mm I.D., SHOWA DENKO) + Shodex GPC KF-401HQ (250 mm × 4.6 mm mm I.D., SHOWA DENKO)	
Column temperature	40°C	
Eluent	Tetrahydrofuran	
Flow rate	0.2 mL/min.	
Injection volume	20 μL	

c) Preparation of standard solution and calculation of concentration

The test sample [100 mg (82.7 mg of test item)] was accurately weighed by an electronic analytical balance and dissolved in tetrahydrofuran in order to obtain 4140 mg/L solution of the test item. The standard solution of 1240 mg/L was then prepared from this solution by dilution with tetrahydrofuran. The test sample was handled under the yellow fluorescent light.

The concentration of the test item in the sample for HPLC analysis was calculated proportionally by comparing the peak area on the chromatogram of the sample for HPLC analysis with that on the chromatogram of 1240 mg/L standard solution (see Table-3 and Fig. 4).

The lowest detectable peak area of the test item was regarded as 1000000 μV·sec considering the noise level, which corresponded to the test item concentration of 9.7 mg/L.

13.5.3 Recovery test

Two test solutions (water + test item), two test solutions (sludge + test item), and a test solution (control blank) were prepared according to the methods described in Section 13.2. These test solutions were pretreated in accordance with the method described in Section 13.5.1, after stirring at room temperature for about 30 minutes. Then, the samples for HPLC analysis obtained in the pretreatment were analyzed according to the analytical conditions described in Section 13.5.2 and the recovery rates of the test item were calculated. The test sample [30 mg (25 mg of test item)] was added in recovery test. The pretreatment was performed under the yellow fluorescent light.

The recovery rates and their averages are shown below (see Table-2 and Fig. 3). The average in each test solution was 90% and more, and the difference between replicate values in each was within 5%. In addition, no peak exceeding the lowest detectable peak area appeared around the peak of the test item on the chromatogram of the test solution (control blank). Therefore, the validity of the pretreatment applied in this study was confirmed.

The concentrations of the test item in the analysis samples were corrected using the averages.

Recovery rate in the test solutions (water + test item)	97.0%, 96.5%	average	96.8%
Recovery rate in the test solutions (sludge + test item)	95.9%, 96.5%	average	96.2%

13.6 Calculation of percentage biodegradation

The percentage biodegradations were calculated by the following equations and rounded off to the whole number.

a) Percentage biodegradation by BOD

$$\text{Percentage biodegradation (\%)} = \frac{\text{BOD} - \text{B}}{\text{TOD}} \times 100$$

BOD : Biochemical oxygen demand in the test solution (sludge + test item) (experimental: mg)

B : Biochemical oxygen demand in the test solution (control blank) (experimental: mg)

TOD : Theoretical oxygen demand required when the test item was completely oxidized (theoretical: mg)

TOD was calculated from the molecular formula $C_{36}H_{32}N_2O_7S$ which was derived from the structural formula described in Section 12.1 b); in case of n=1, R^1=H-, CH_3O- and $C_{10}H_5N_2O_3S$-, R^2= H-, H- and CH_3-. The oxidization form of the nitrogen was ammonia in the calculation of TOD.

b) Percentage biodegradation of test item

$$\text{Percentage biodegradation (\%)} = \frac{\text{Sw} - \text{Ss}}{\text{Sw}} \times 100$$

Ss : Residual amount of the test item in the test solution (sludge + test item) (experimental: mg)

Sw : Residual amount of the test item in the test solution (water + test item) (experimental: mg)

13.7 Treatment of numerical values

Values were rounded off in accordance with JIS Z 8401:1999 rule B. The atomic weight of each element used in this study was based on the 4-digit atomic weight table 2012 provided by The Chemical Society of Japan.

14. Validity of test conditions

The validity criteria of the tests and the values in this test are shown in the following table. This test was judged to be valid because all the values in this test met the criteria. This test was deviated from part of the test method, because the concentration of the test item (83.3 mg/L) in this test was lower than that provided in the test method (100 mg/L). However it was judged that the evaluation of the biodegradability of the test item was possible. In calculation of TOD of aniline, ammonia was adopted as the oxidization form of the nitrogen.

		Value in this test	Value of criterion	Table
Difference between extremes of replicate values of percentage biodegradation	Percentage biodegradation by BOD	4%	< 20%	1
	Percentage biodegradation of test item	3%		3
Percentage biodegradation of aniline by BOD	After 7 days	79%	> 40%	1
	After 14 days	93%	> 65%	

15. Factors that affected reliability of test

No adverse effects on the reliability of this test were noted.

16. Results and discussion

16.1 Appearances of test solutions

Appearances of test media in cultivation vessels were as follows.

	Test solution	Appearance (visual)	pH
At the start of cultivation	Water + test item	The test item was not dissolved. The test solution was colorless.	-
	Sludge + test item	The test item was not dissolved. The test solution was colorless.	-
At the end of cultivation	Water + test item	Insoluble compound was observed. The test solution was colorless.	Vessel No. 5 6.4
	Sludge + test item	Insoluble compound in addition to the sludge was observed. Growth of the sludge could not be judged. The test solution was white turbid.	Vessel No. 1 7.2 Vessel No. 2 7.3 Vessel No. 3 7.3

16.2 Analytical results of test solutions

Analytical results of the test solutions after 28 days were as follows.

		Water + test item	Sludge + test item			Theoretical amount	Table	Fig.
		Vessel No. 5	Vessel No. 1	Vessel No. 2	Vessel No. 3			
BOD*1	mg	0	8.1	6.1	7.3	50.3*2	1	1
Residual amount and percentage residue of test item (HPLC)	mg	24.4	24.1	24.3	23.6	25.0	3	4
	%	98	96	97	94	-		

*1 The value of the test solution (control blank) was subtracted from the values of the test solutions (sludge + test item).

*2 TOD was calculated from the molecular formula $C_{36}H_{32}N_2O_7S$ which was derived from the structural formula described in Section 12.1 b); in case of n=1, R^1=H-, CH_3O- and $C_{10}H_5N_2O_3S$-, R^2= H-, H- and CH_3-. The oxidization form of the nitrogen was ammonia in the calculation of TOD.

16.3 Percentage biodegradation

Percentage biodegradations after 28 days were as follows.

		Sludge + test item				Table
		Vessel No. 1	Vessel No. 2	Vessel No. 3	Average	
Percentage biodegradation by BOD	%	16	12	15	14	1
Percentage biodegradation of test item (HPLC)	%	1	0	3	2	3

16.4 Discussion

a) Results of test item analysis

The percentage residue of the test item was 98% in the test solution (water + test item) and 94-97% in the test solutions (sludge + test item). In addition, no peaks corresponding to converted products were detected in the analyses of the test item and the low-molecular-weight components of the test item (see fig. 3 and Annex fig. 1). From the above-mentioned results, it was considered that the test item was not biodegraded and remained under the test conditions of this study.

b) Percentage biodegradation by BOD

The average percentage biodegradation by BOD was 14%, showed that the test item was biodegraded by microorganisms.

Considering that the test item remained in the test solution, the percentage biodegradation by BOD was presumed to be derived from biodegradation of methyl isobutyl ketone (MIBK) [METI number (2)-542] contained as impurity in the test sample. If all amount of MIBK added to the test solution is biodegraded, the percentage biodegradation by BOD is calculated as 14.1 mgO_2 and higher than measured value of BOD (6.1-8.1 mgO_2). Therefore, it is presumed that some of MIBK added to the test solution was biodegraded under the conditions of this test.

MIBK: $C_6H_{12}O + 8.5O_2 \rightarrow 6CO_2 + 6H_2O$

Calculated value of BOD:

30 (mg) × 17.3 (content percentage of MIBK) / 100 × $8.5O_2$ / $C_6H_{12}O$ = 14.1 (mgO_2)

c) Deviated from the test method

After experimental completion, the purity of the test item was modified by sponsor. Therefore this study could not be performed under the concentration of the test item 100 mg/L prescribed in the test method. However, the concentration of the test item after the modification of the purity of the test item was 83.3 mg/L and it was judged that the evaluation of the biodegradability of the test item was possible.

17. Conclusion

The test item was not biodegraded under the test conditions of this study.

18. Remarks

18.1 Instruments used for test

Fourier transform infrared spectrophotometer :	Shimadzu type IRPrestige-21
Closed system oxygen consumption measuring apparatus :	see page 12
High-performance liquid chromatograph :	see page 14
Electronic analytical balance :	Sartorius type CP224S
	Sartorius type ME235P
pH meter :	DKK-TOA type HM-50G
Shaker :	Taitec type SR-2w
Rotary evaporator :	Tokyo Rika Kikai type N-1000V

18.2 Reagents used for analysis

Tetrahydrofuran (HPLC grade) :	Kanto Chemical
Ethyl acetate (reagent grade) :	Kanto Chemical
Sodium chloride (reagent grade) :	MANAC
Phosphoric acid (reagent grade) :	Wako Pure Chemical Industries

Table 1 Calculation table for percentage biodegradation by BOD

Study No. 15857 Duration of incubation: 28 days

Vessel No.	7th day		14th day		21st day		28th day		Average
	BOD(mg)	Deg.(%)	BOD(mg)	Deg.(%)	BOD(mg)	Deg.(%)	BOD(mg)	Deg.(%)	Deg.(%)
[5]	0.0	-	0.0	-	0.0	-	0.0	-	-
[1]	9.9	11	13.2	13	14.8	15	16.1	16	
[2]	8.4	8	11.4	10	13.0	11	14.1	12	14
[3]	9.6	10	12.8	13	14.2	14	15.3	15	
[4]	4.4	-	6.4	-	7.3	-	8.0	-	-
[6]	61.2	79	74.0	93	76.6	96	77.7	96	-

Deg. : Percentage biodegradation

Water + test item : Vessel No. [5]
Sludge + test item : Vessel No. [1][2][3]
Control blank : Vessel No. [4]
Sludge + aniline : Vessel No. [6]

Additive amount of test item : 25 mg
Additive amount of aniline : 30 mg

Deg. = (BOD - B)/(TOD) × 100

B : BOD of control blank

TOD of test item : 50.3 mg

$C_{36}H_{32}N_2O_7S + 40.00\,O_2 \rightarrow 36.00\,CO_2 + 13.00\,H_2O + SO_2 + 2.00\,NH_3$

TOD = additive amount of test item ×

$40.00\,O_2 / C_{36}H_{32}N_2O_7S = 50.3$

TOD of aniline : 72.3 mg

$C_6H_7N + 7.00\,O_2 \rightarrow 6.00\,CO_2 + 2.00\,H_2O + NH_3$

TOD = additive amount of aniline × $7.00\,O_2 / C_6H_7N = 72.3$

See Fig. 1

Dec. 25, 2012 Name Ayaka Maeda

確認 12.12.25 奥園

Table-2 Calculation table for recovery rate of test item

Study No. 15857

Sample description	A	D	E	F
Standard solution 1240 mg/L	124882024			
Water + test item -1	122162829	24.3	97.0	96.8
Water + test item -2	121448961	24.1	96.5	
Sludge + test item -1	120786511	24.0	95.9	96.2
Sludge + test item -2	121542396	24.1	96.5	
Control blank	n.d.			

Amount of test item added : 25 (mg)

A : Peak area (μV·sec)

B : Final volume : 10 (mL)

C : Ratio of portion used for analysis : 300/300×50/100

D : Recovery amount (mg)

$Dw = G \times (A(\text{Water + test item}) / A(\text{Standard})) \times (B / C) / 1000$

$Ds = G \times \{ (A(\text{Sludge + test item}) - A(\text{Control blank})) / A(\text{Standard}) \} \times (B / C) / 1000$

E : Recovery rate (%)

$E = D / 25 \text{ (mg)} \times 100$

F : Average recovery rate (%)

G : Concentration of standard solution : 1240 (mg/L)

See Fig. 3

December 20, 2012 Name Ayaka Maeda

Table-3 Calculation table for percentage biodegradation of test item

Study No. 15857

Sample description	A	E	F	G	H
Standard solution 1240 mg/L	127477045				
[5] Water + test item	121367119	24.4	98		
[1] Sludge + test item	119267555	24.1	96	1	
[2] Sludge + test item	120265289	24.3	97	0	2
[3] Sludge + test item	116739599	23.6	94	3	
[4] Control blank	n.d.				

Amount of test item added : 25 (mg)

A : Peak area (μV·sec)

B : Final volume : 10 (mL)

C : Ratio of portion used for analysis : 300/300×50/100

D : Recovery rate : 96.8 (%) (Water + test item)
96.2 (%) (Sludge + test item)

E : Residual amount of test item (mg)

Ew = I × (A(Water + test item) / A(Standard)) × (B / C) / (D / 100) / 1000

Es = I × { (A(Sludge + test item) - A(Control blank)) / A(Standard) }
× (B / C) / (D / 100) / 1000

F : Percentage residue (%)

F = E / 25 (mg) × 100

G : Percentage biodegradation (%)

G = { (E(Water + test item) - E(Sludge + test item))
/ E(Water + test item) } × 100

H : Average percentage biodegradation (%)

I : Concentration of standard solution : 1240 (mg/L)

See Fig. 4

December 20, 2012 Name Ayaka Maeda

Study No. 15857 (Test item RESIN-A2M)

Cultivation conditions:

Concentration

Test item ..... * ~~100~~ 83.3 (mg/L)

Reference item aniline ..... 100 (mg/L)

Activated sludge ..... 30 (mg/L)

Temperature ..... 25 ± 1 ℃

Duration ..... 28days (Aug.07,2012 - Sep.04,2012)

Note: –

Vessel No.	Sample Description	BOD (mg)			
		7th day	14th day	21st day	28th day
[1]	Sludge + test item	9.9	13.2	14.8	16.1
[2]	Sludge + test item	8.4	11.4	13.0	14.1
[3]	Sludge + test item	9.6	12.8	14.2	15.3
[4]	Control blank [B]	4.4	6.4	7.3	8.0
[5]	Water + test item	0.0	0.0	0.0	0.0
[6]	Sludge + aniline	61.2	74.0	76.6	77.7

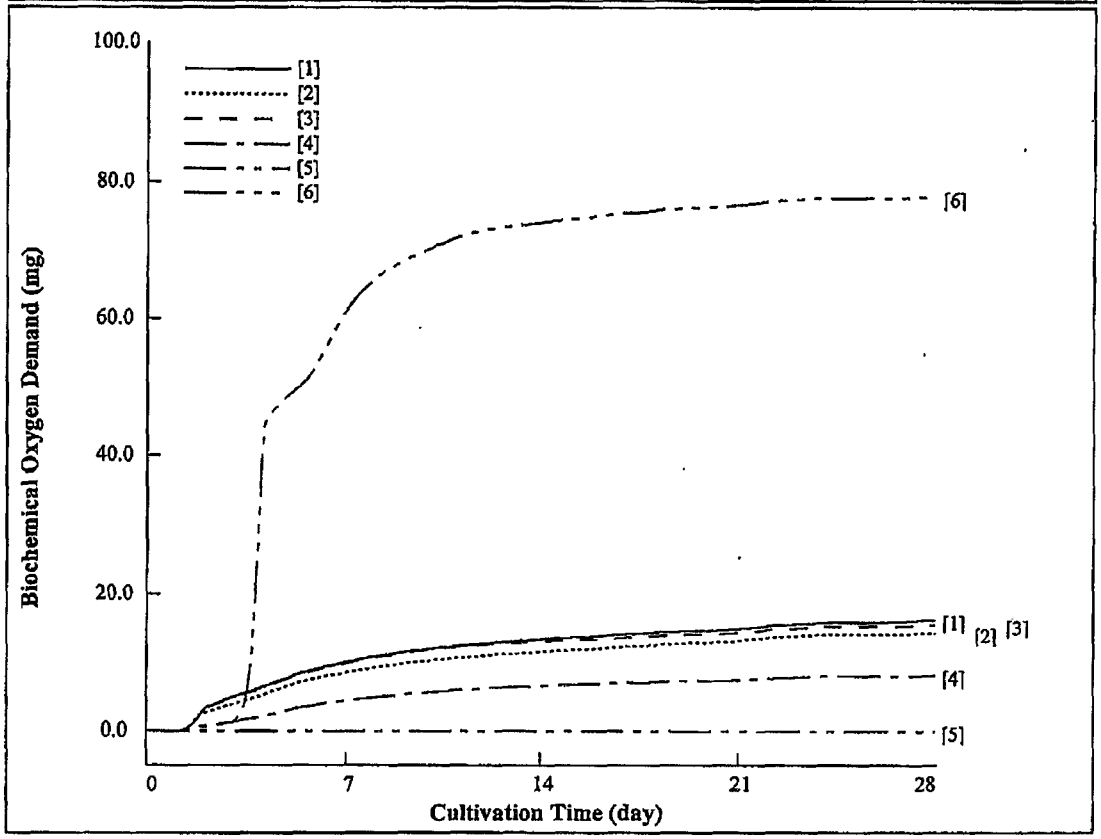


Fig. 1 Chart of BOD.

Sep.04,2012 Name Ayaka Maeda

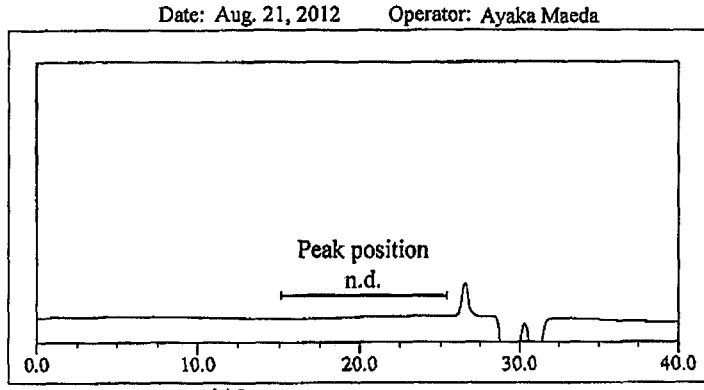
* 被験物質純度変更のため訂正
2012.12.20 前田

確認 '12.12.20 奥園

確認 '12.9.-5 奥園

Solvent blank

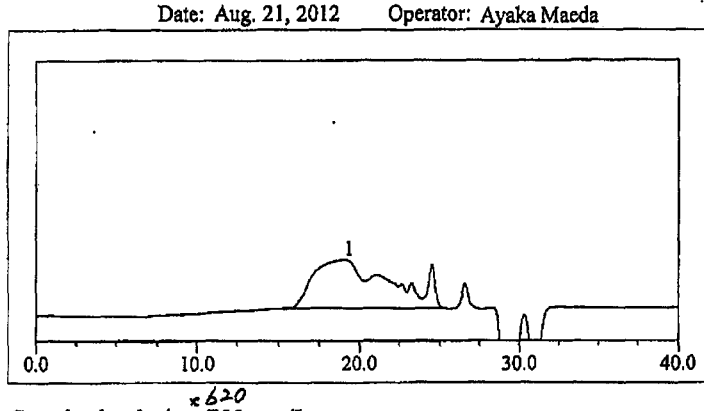
Date: Aug. 21, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	-	-	-	-
Total	-	-	0	0.00

Standard solution ~~375~~ 310 mg/L

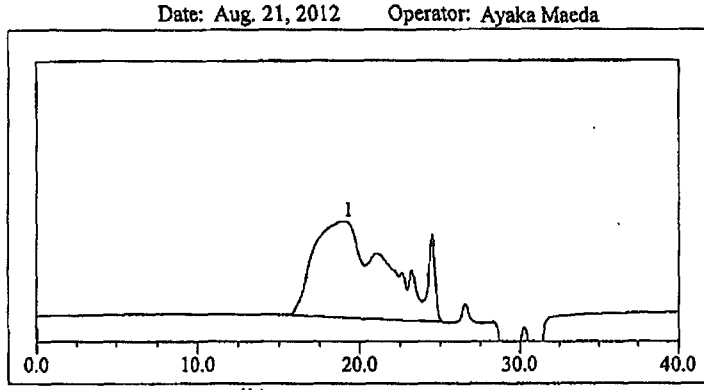
Date: Aug. 21, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	19.05	95634	31001891	100.00
Total	-	-	31001891	100.00

Standard solution ~~750~~ 620 mg/L

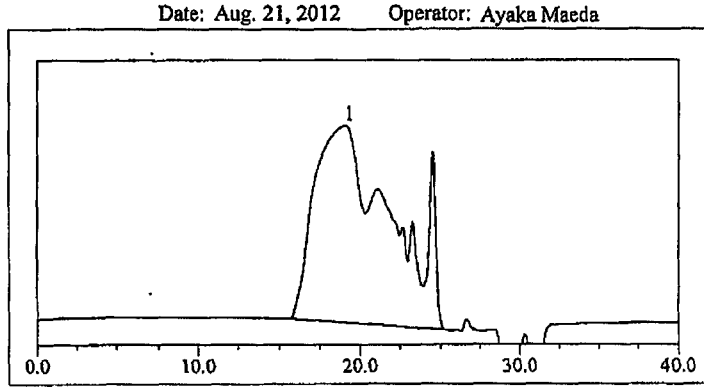
Date: Aug. 21, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	18.98	190123	61567212	100.00
Total	-	-	61567212	100.00

Standard solution ~~1500~~ 1240 mg/L

Date: Aug. 21, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	19.01	382812	124469535	100.00
Total	-	-	124469535	100.00

確認 12.8.22 奥園

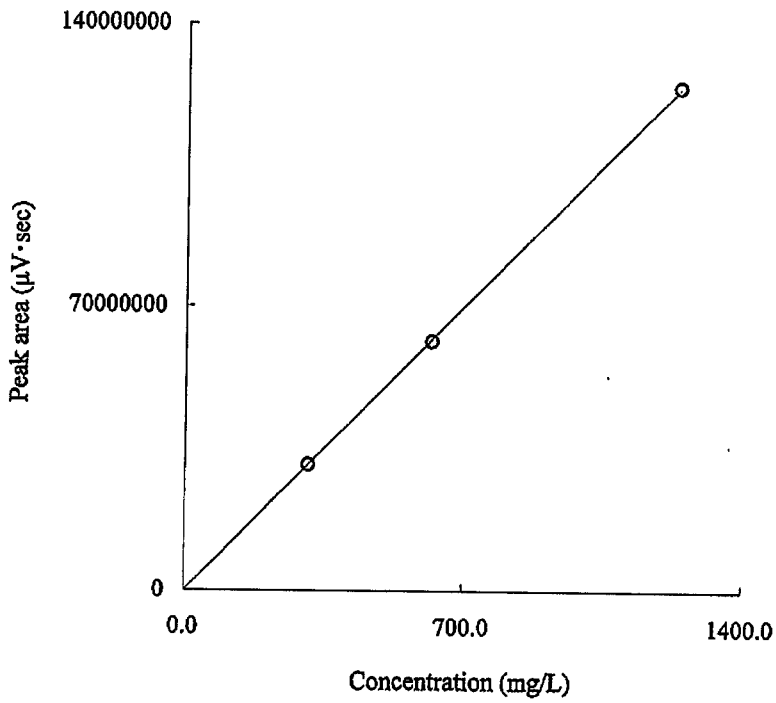
Fig.2 - 1 Chromatograms of HPLC analysis for calibration curve.

＊3ヶ所被験物質純度変更のため訂正

2012. 12. 20 前田

確認 121220 奥園

Date 2012. 8. 22 Name Ayaka Maeda



y = 100156x
r = 1.00

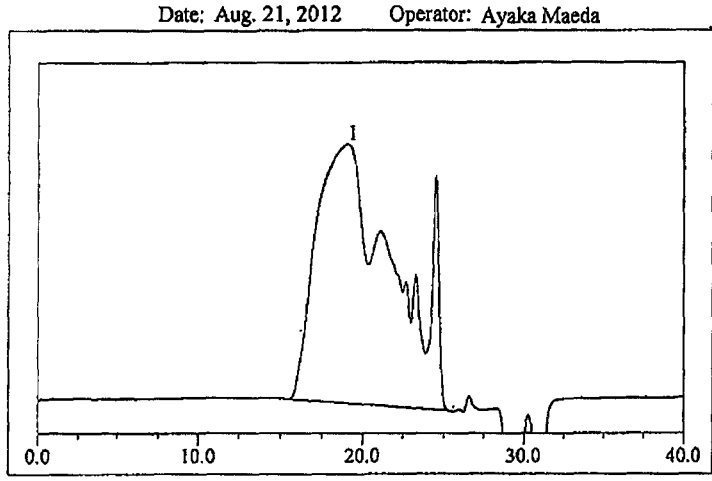
Concentration (mg/L)	Peak area (μV·sec)
310	31001891
620	61567212
1,240	124469535

Fig. 2 - 2 Calibration curve of test item.

December 20, 2012 Name Ayaka Maeda

Standard solution *1240 ~~1500~~ mg/L

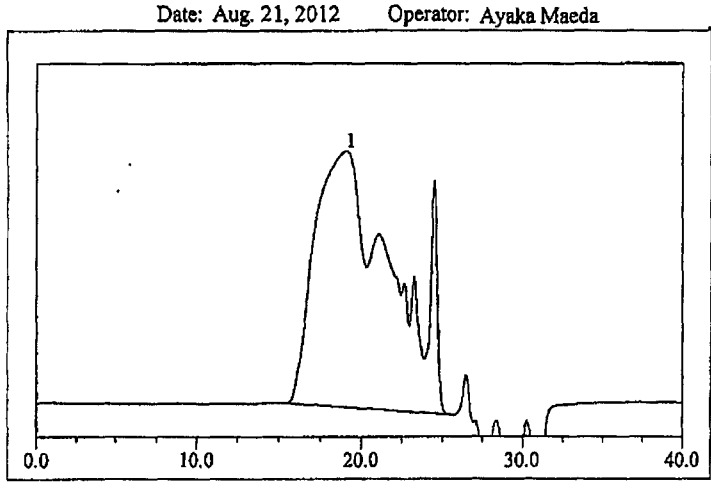
Date: Aug. 21, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	19.05	383938	124882024	100.00
Total	-	-	124882024	100.00

Water + test item - 1

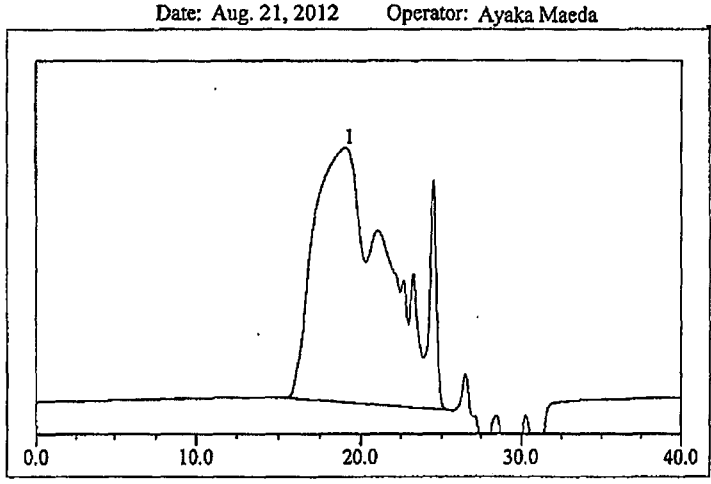
Date: Aug. 21, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	19.02	375024	122162829	100.00
Total	-	-	122162829	100.00

Water + test item - 2

Date: Aug. 21, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	19.03	372898	121448961	100.00
Total	-	-	121448961	100.00

Fig. 3 - 1 Chromatograms of HPLC analysis for recovery test.

* 被験物質純度変更の訂正
2012.12.20 前田

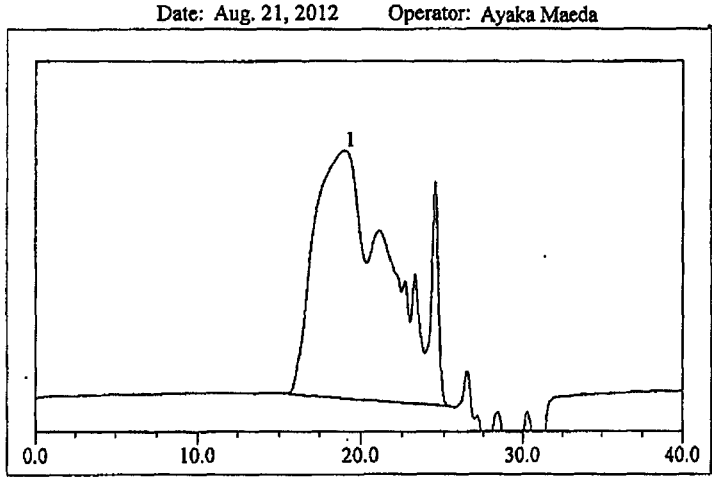
確認 12.12.20 奥園

Date 2012.8.22 Name Ayaka Maeda

確認 12.8.22 奥園

Sludge + test item - 1

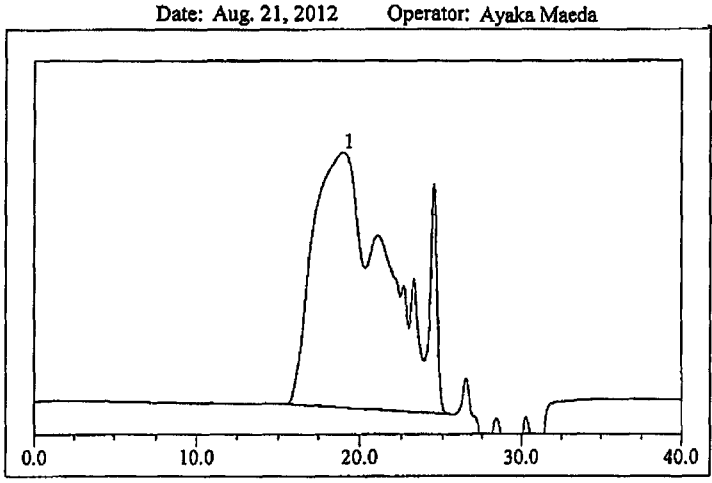
Date: Aug. 21, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	19.09	372226	120786511	100.00
Total	-	-	120786511	100.00

Sludge + test item - 2

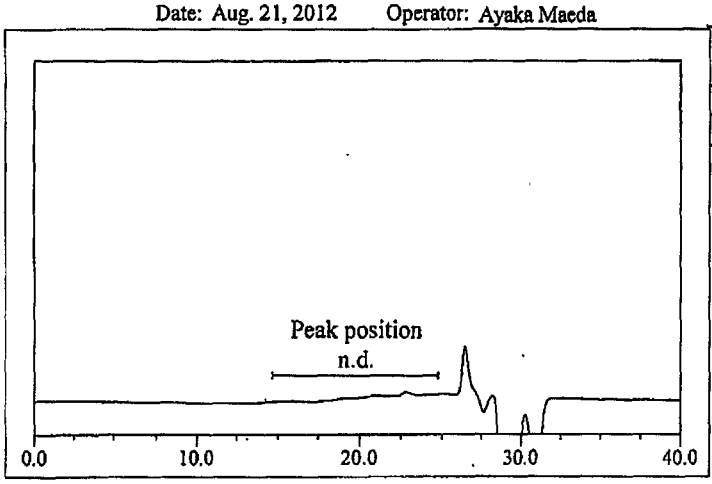
Date: Aug. 21, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	19.05	374602	121542396	100.00
Total	-	-	121542396	100.00

Control blank

Date: Aug. 21, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	-	-	-	-
Total	-	-	0	0.00

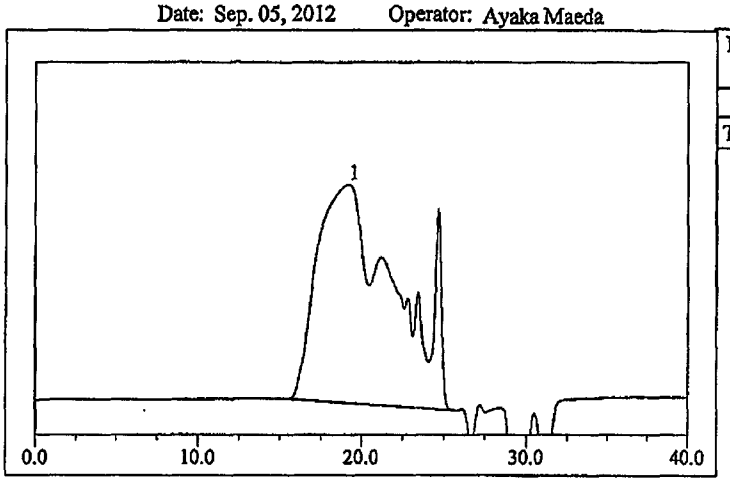
Fig. 3 - 2 Chromatograms of HPLC analysis for recovery test.

Date 2012.8.22 Name Ayaka Maeda

確認 12.8.22 奥園

Standard solution 1240 ~~1500~~ mg/L

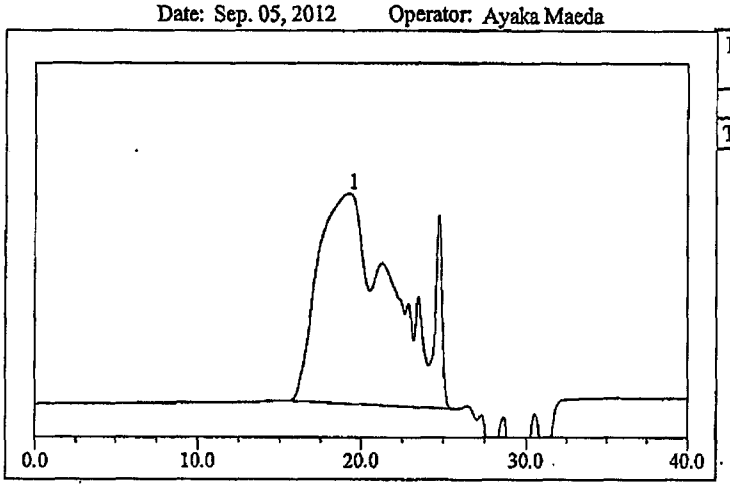
Date: Sep. 05, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	19.20	388182	127477045	100.00
Total	-	-	127477045	100.00

[5] Water + test item

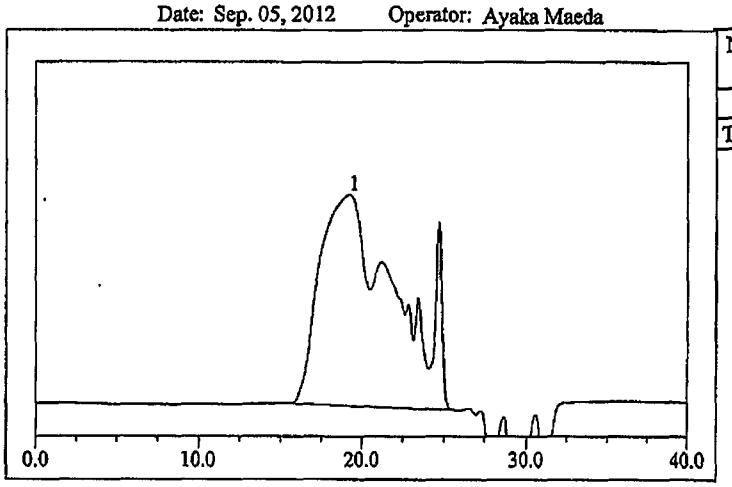
Date: Sep. 05, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	19.19	370115	121367119	100.00
Total	-	-	121367119	100.00

[1] Sludge + test item

Date: Sep. 05, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	19.19	370674	119267555	100.00
Total	-	-	119267555	100.00

Fig. 4 - 1 Chromatograms of HPLC analysis for test solution.

＊被験物質純度変更のため訂正
2012.12.20 前田

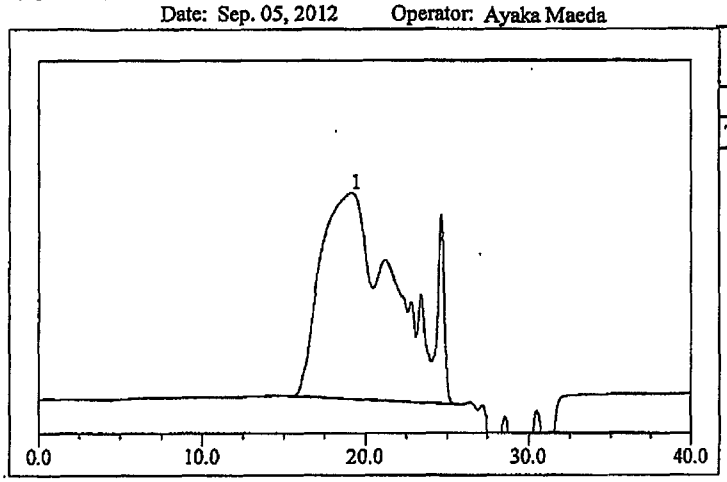
確認 121220 奥園

Date 2012.9.5 Name Ayaka Maeda

確認 '12.9.-5 奥園

[2] Sludge + test item

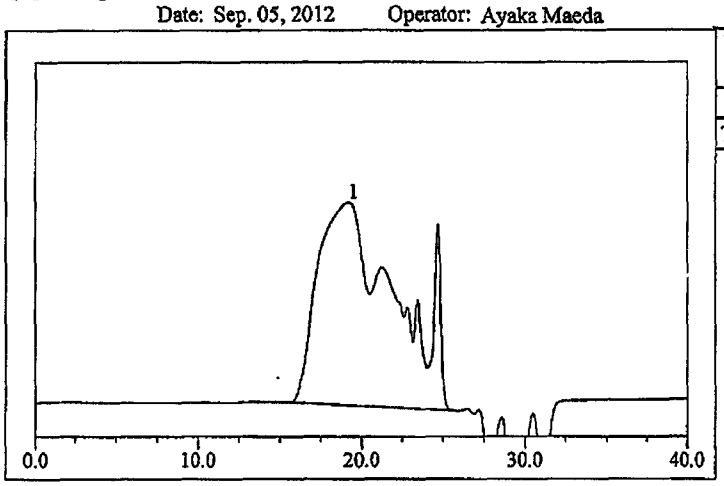
Date: Sep. 05, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	19.13	367181	120265289	100.00
Total	-	-	120265289	100.00

[3] Sludge + test item

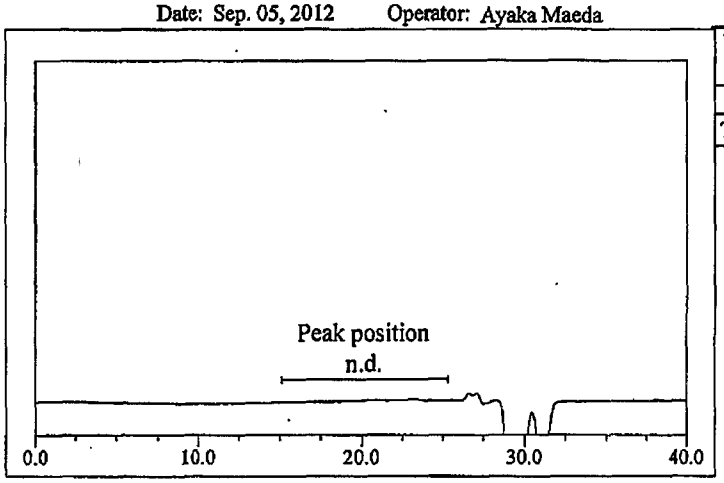
Date: Sep. 05, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	19.19	359255	116739599	100.00
Total	-	-	116739599	100.00

[4] Control blank

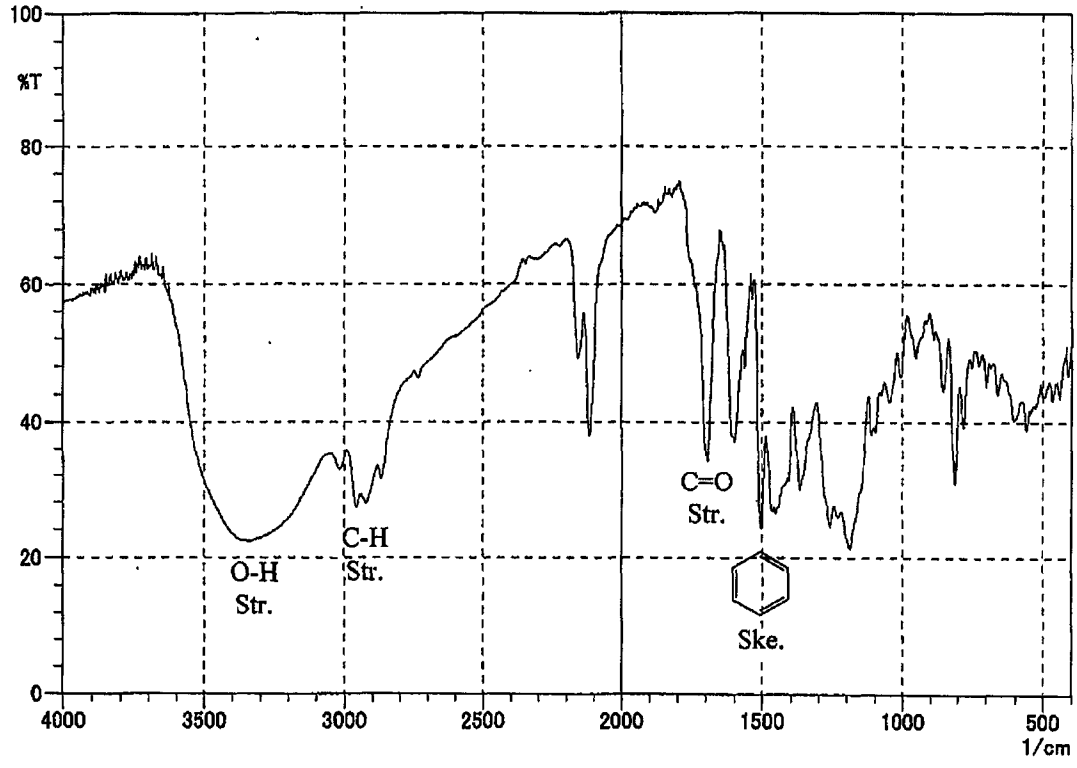
Date: Sep. 05, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μV)	Area (μV·sec)	Area (%)
1	-	-	-	-
Total	-	-	0	0.00

Fig. 4 - 2 Chromatograms of HPLC analysis for test solution.

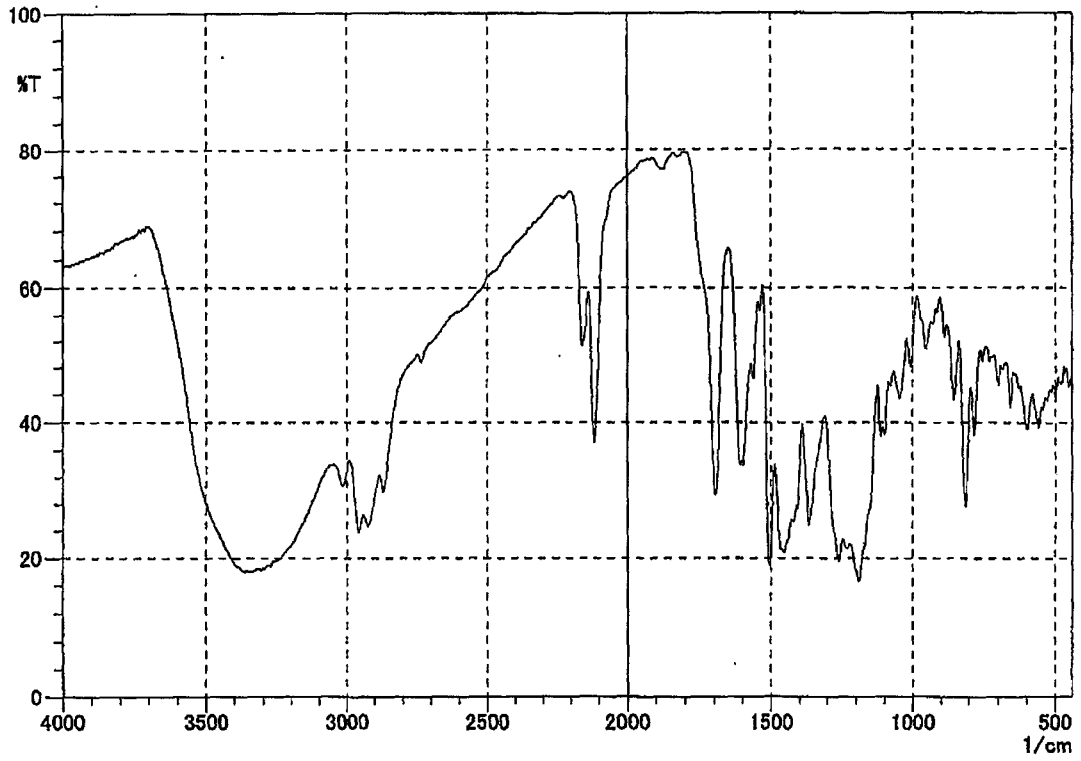
Date 2012. 9. 5 Name Ayaka Maeda



Instrument	: Shimadzu IRPrestige-21
Study No.	: 15857
Sample	: Test item
Method	: KBr tablet
Date	: May 28, 2012
Name	: Ayaka Maeda

Fig. 5 - 1 IR spectrum of test item measured before experimental start.

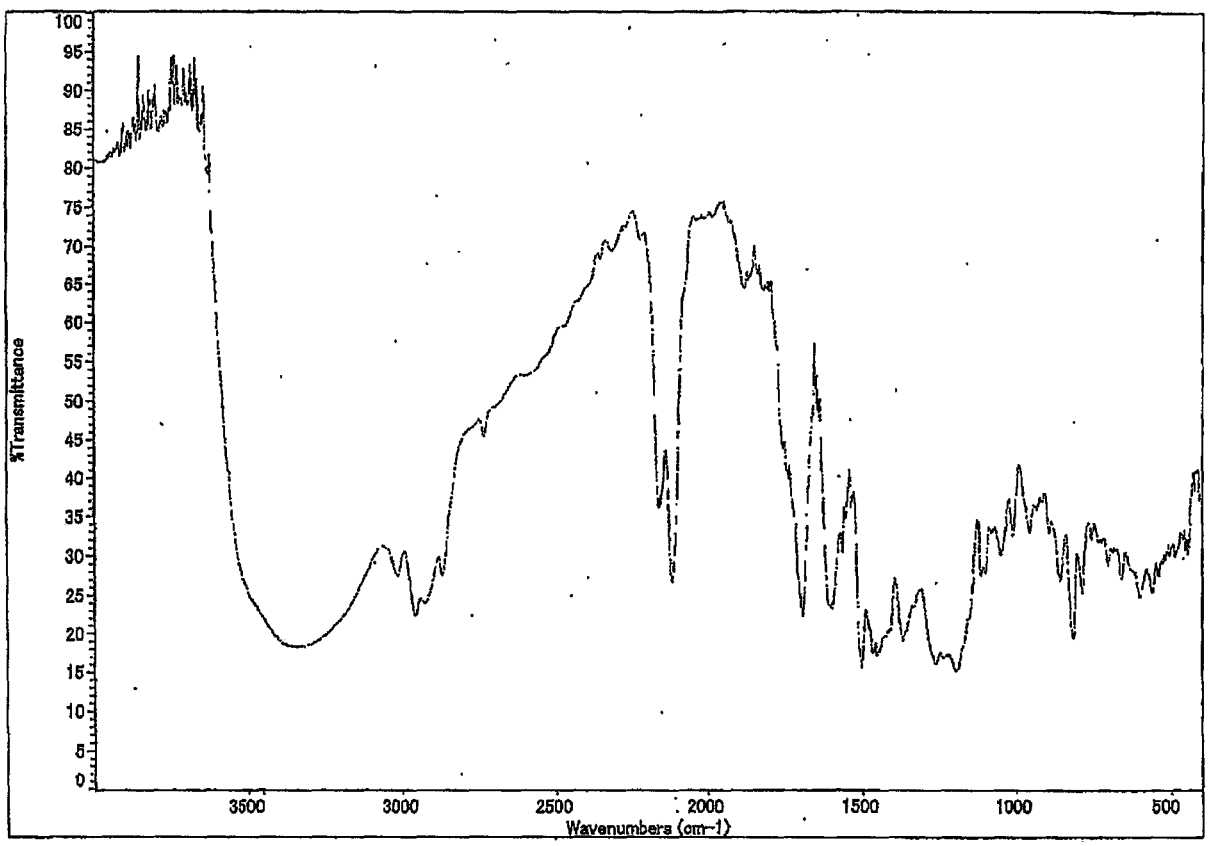
確認
'12.5.29
英國



Instrument	: Shimadzu IRPrestige-21
Study No.	: 15857
Sample	: Test item
Method	: KBr tablet
Date	: November 16, 2012
Name	: Ayaka Maeda

Fig. 5 - 2 IR spectrum of test item measured after experimental completion.

確認 7211.16 典図



Reference 1 IR spectrum supplied by sponsor.

Annex 1

Qualitative analysis of low-molecular-weight components of test item

1. Objective

There was no significant change of the test item on the quantitative analysis of the test item. In order to confirm the conversion of low-molecular-weight components of the test item in detail, low-molecular-weight components of the test item in the test solutions were analyzed with HPLC by using reverse-phase chromatography column. In general, the column is suited for the analysis of the region from low-molecular-weight to oligomer.

2. Method of analysis

The samples for HPLC analysis prepared in Section 13.5.1 of final report were analyzed. The conversion of the test item in the sample for HPLC analysis was confirmed by comparing the peak shape obtained on the chromatogram of the sample for HPLC analysis with that of a solution of the test item.

a) Analytical conditions

Instrument	High-performance liquid chromatograph	
Pump	LC-20AD	(Shimadzu)
UV-VIS detector	SPD-20AV	(Shimadzu)
Column oven	CTO-20AC	(Shimadzu)
Auto injector	SIL-20A	(Shimadzu)
System controller	SCL-10Avp	(Shimadzu)
Degasser	DGU-20A3	(Shimadzu)
Column	L-column ODS (150 mm × 2.1 mm I.D., particle size 5 μm, Chemicals Evaluation and Research Institute)	
Column temperature	40°C	
Eluent	A : Tetrahydrofuran	
	B : Ultrapure water	
	Gradient condition	

Time (min)	A (%)	B (%)
0.0	30	70
14.0	100	0
24.0	100	0

Flow rate	0.2 mL/min
Measurement wavelength	282 nm
Injection volume	2 μL

b) Preparation of solution of test item

The standard solution prepared in Section 13.5.2 c) of final report was used as 1240 mg/L solution of the test item.

3. Result of analysis and discussion

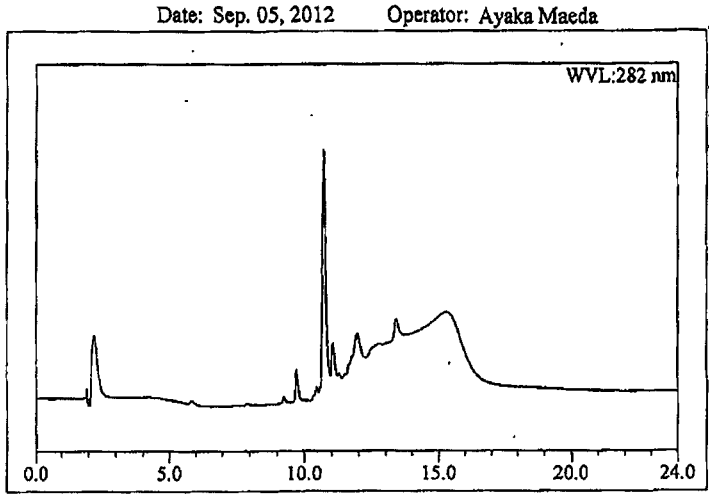
As a result, conversion of the peak shape of low-molecular-weight components of the test item was not observed on HPLC chromatogram, and any converted products were not detected (see Annex fig. 1). Then, it was judged that there was no significant change of low-molecular-weight components of the test item.

4. Supporting data

Annex fig. 1 Chromatograms of HPLC analysis for test solution
(qualitative analysis for low-molecular-weight components of test item)

Standard solution *1240 ~~1500~~ mg/L

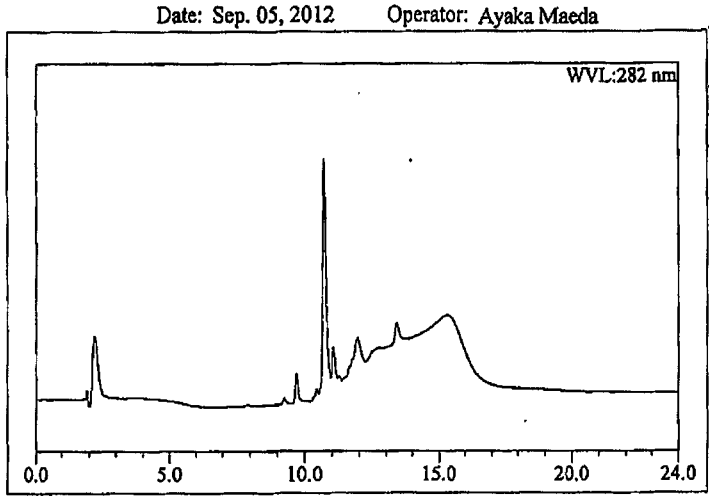
Date: Sep. 05, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μAU)	Area (μAU·sec)	Area (%)
1	-	-	-	-
Total	-	-	0	0.00

[5] Water + test item

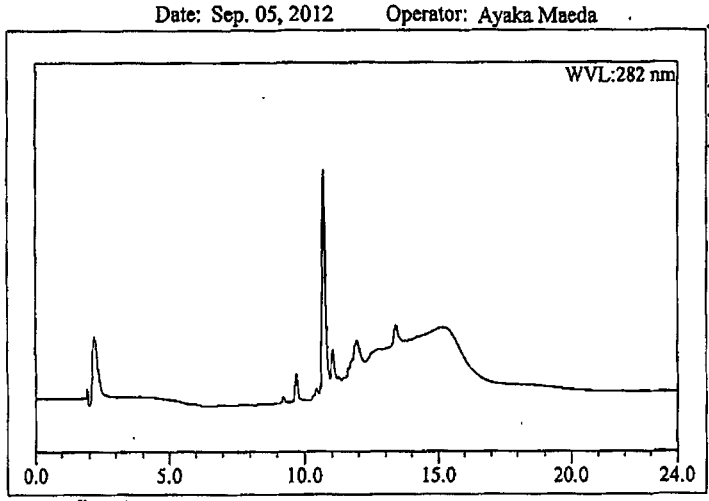
Date: Sep. 05, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μAU)	Area (μAU·sec)	Area (%)
1	-	-	-	-
Total	-	-	0	0.00

[1] Sludge + test item

Date: Sep. 05, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μAU)	Area (μAU·sec)	Area (%)
1	-	-	-	-
Total	-	-	0	0.00

Annex fig. 1 - 1 Chromatograms of HPLC analysis for test solution (qualitative analysis for low-molecular-weight components of test item).

* 被験物質純度変更の訂正 Date 2012.9.5 Name Ayaka Maeda

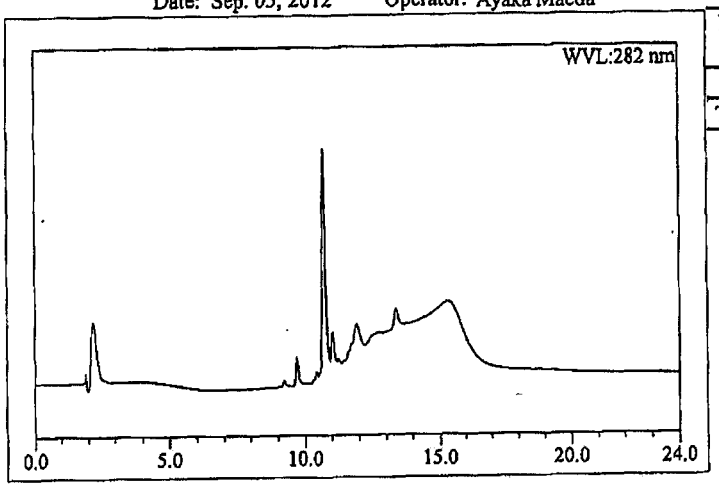
2012.12.20 前田

確認 '12.12.20 奥園

確認 '12.9.-5 奥園

[2] Sludge + test item

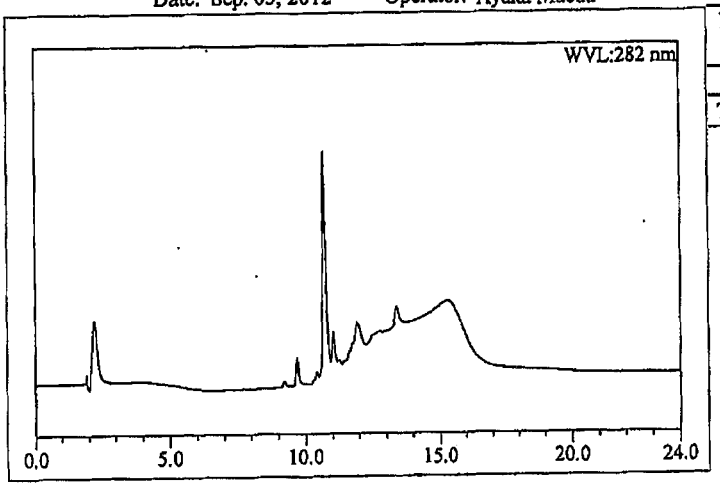
Date: Sep. 05, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μAU)	Area (μAU·sec)	Area (%)
1	-	-	-	-
Total	-	-	0	0.00

[3] Sludge + test item

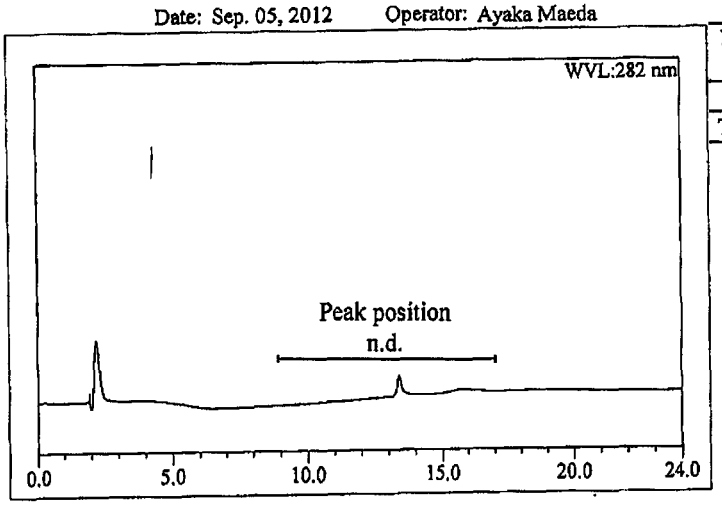
Date: Sep. 05, 2012 Operator: Ayaka Maeda



No.	Time (min)	Height (μAU)	Area (μAU·sec)	Area (%)
1	-	-	-	-
Total	-	-	0	0.00

Control blank

Date: Sep. 05, 2012 Operator: Ayaka Maeda



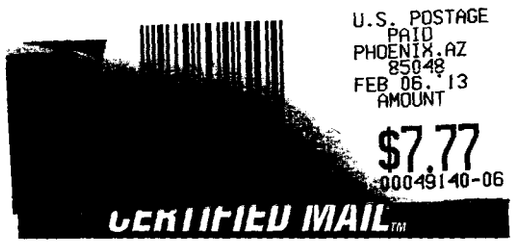
No.	Time (min)	Height (μAU)	Area (μAU·sec)	Area (%)
1	-	-	-	-
Total	-	-	0	0.00

Annex fig. 1 - 2 Chromatograms of HPLC analysis for test solution (qualitative analysis for low-molecular-weight components of test item).

Date 2012.9.5 Name Ayaka Maeda

確認 '12.9.-5 興國

Shin-EtsuMicroSi®
MICROELECTRONIC MATERIALS
10028 S. 51st Street • Phoenix, AZ 85044



7010 3090 0000 6260 0804